DEVELOPMENT OF NEUTRON ACTIVATION ANALYSIS PROCEDURES FOR THE DETERMINATION OF OXYGEN IN POTASSIUM

Third Quarterly Report

by

E. L. Steele

Prepared For

National Aeronautics and Space Administration
Lewis Research Center
Under Contract NAS3-2537

GENERAL ATOMIC
DIVISION OF
GENERAL DYNAMICS

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NASA-CR-54099
GA-5339

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(Period Ending March 31, 1964)

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Sponsored by
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Lewis Research Center

Technical Management
NASA-Lewis Research Center
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R. A. Lindberg

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John Jay Hopkins Laboratory for Pure and Applied Science
P.O. Box 608, San Diego, California

Contract: NAS3-2537
Issued: June 19, 1964

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I. INTRODUCTION

The development of neutron activation analysis procedures for the determination of oxygen in potassium has been continued along the lines described in Section V of Report GA-4855. The principal effort during this quarter was devoted to improvements in the equipment. Particular attention was given to the problem of neutron monitoring during irradiation. The sample transfer system was upgraded to take advantage of the short-half-life radioactive N^{16} product. All manual operations, including transfer, timing, analyzer starts and stops, accelerator on and off, and neutron flux determinations, were eliminated by an automatic control mechanism which steps the system through the entire procedure. A new device was designed and installed to ensure a more uniform sample irradiation.

Further development work was done on sample handling and encapsulation. A variety of metals and alloys were examined for residual oxygen content, and several cleaning techniques were investigated.

Improvements were made in the detection system. The preamplifier-stage time constants were shortened to reduce pulse "pileup" from large amounts of radioactivity generated in the capsule and sample matrix. A new detector, which measures the high-energy beta particles from N^{16} rather than gamma rays, was also investigated.

II. ANALYTICAL SYSTEM IMPROVEMENTS

The analytical results described in GA-4855 were obtained with a completely manual system. That is, all irradiation and transfer times were read from a stopwatch and the equipment was operated by hand. Relative neutron fluxes were integrated yields over the entire irradiation time. The level of precision in the 10- to 1000-mg oxygen region was 2%. The sensitivity in photopeak counts per gram of oxygen was approximately 10^5. Since the intent of the contract was to increase the general precision of the method as well as the sensitivity, it was essential that the procedure be automated and that a neutron flux measurement that more nearly described the irradiation conditions be developed.

DETECTOR SYSTEMS

Interference in the detection of the 6-Mev gamma rays from N^{16} is not generally considered a problem. However, the fact that many elements have either a higher activation cross section than oxygen or a greater abundance in the sample, or both, often leads to a large amount of activity in addition to and relative to the N^{16} resulting from oxygen activation. The interfering activities may cause an inordinate amount of dead time in the measuring device, which seriously affects the counting statistics of the short-lived N^{16}. In addition, because of the relatively slow resolution time of the sodium iodide scintillation crystal ($\sim 10^{-7}$ sec), it is possible for two or more gamma rays from a very radioactive source to be seen "simultaneously" and added prior to pulse-height analysis. Such "pileup" may raise the base of the N^{16} photopeak, which worsens the statistics of counting or obscures the photopeak altogether.

Two different techniques were used to minimize the potential "pileup." First, a redesigned preamplifier, which accepts the signal from the photomultiplier tube, was tried. The new design has a resolution time of 1 μsec. This is four times faster than the original device. The chance for "pileup" was reduced, then, by a factor of four. Electronic "deadtime" was also reduced, making the entire system linear over a much larger concentration range.

The second technique applied to this problem was the use of a Cerenkov detector.[1] Unlike the sodium iodide scintillation crystals previously used, this device measures the Cerenkov radiation from high-energy beta particles as a function of their energy. In general, when a charged particle traverses a path, ℓ, in a medium, the number of photons radiated, N, along a short segment, $d\ell$, is given by

$$N = K \int_{\beta=\beta(\max)}^{1/\eta} 1 - \frac{1}{\beta^2 \eta^2} \, d\ell ,$$

where β is the velocity of the particle relative to the velocity of light in a vacuum, η is the refractive index of the medium, $1/\eta$ is the minimum velocity for the Cerenkov effect, and K is a combined constant that includes the fine structure constant and the radiation frequency range under consideration.

From the beta-particle energy sufficient to cause Cerenkov radiation in water (0.26 Mev), the photon output increases rapidly with beta energy up to about 2 Mev. Further increase in beta energy results in a smaller proportionate increase in light output, in accordance with the above equation.

However, the number of beta particles of sufficient energy to cause a large Cerenkov radiation in water or Lucite increases markedly with increasing maximum energy of emission because of the energy-spectrum characteristics of beta emitters. Thus, the Cerenkov detector offers an advantage for measuring beta particles (10.4 Mev E_{max}) from N^{16} in samples containing relatively large amounts of interference in the form of gamma rays or low-energy beta particles. In addition, the resolving time of the detector is of the order of only 10^{-10} sec.

One disadvantage in counting only the high-energy beta particles from the N^{16} isotope is the relatively low abundance of these particles relative to the gamma rays. As seen from the decay scheme of N^{16} (see Fig. 1), ~80% of the disintegrations proceed by emission of relatively low-energy beta particles (E_{max} values in the range of 1.55 to 4.34 Mev) to several excited states of O^{16}. These excited states return to the ground state by gamma-ray emission. The remaining 20% of the decays go directly to the ground state of O^{16} by energetic beta emission (10.4 Mev E_{max}). Thus, there are about four times as many energetic gamma rays as there are highly energetic betas. The nature of beta emission, however, is such that the average beta energy is only about 1/3 of the E_{max}. It is the Cerenkov detector's efficiency for beta particles and insensitivity to gamma rays that makes it better for N^{16} detection than sodium iodide detectors. The fact that the Cerenkov detector is cheaper and more rugged is a further advantage.

SAMPLE HANDLING AND ENCAPSULATION EQUIPMENT

A new inert-atmosphere chamber has been installed for handling and encapsulating oxygen-sensitive samples. The unit consists of a hermetically sealed dry box combined with a highly efficient purification system which constantly maintains an inert atmosphere with less than 1 ppm moisture and oxygen. The purification system is basically a copper furnace followed by a soda-lime trap. Materials are inserted into the unit through a vacuum chamber, which is evacuated and back-flushed several times with purified argon. The box is equipped with induction heaters for outgassing, balances for weighing samples, and arc welding torchs for encapsulation. A MAGNEFORM* head will be installed for magnetically sealing copper and aluminum capsules.

ACCELERATOR FACILITY

Construction of a new facility to house the latest model Texas Nuclear Corporation 150-kv positive-ion Cockcroft-Walton accelerator is nearing

*Registered in U. S. Patent Office.

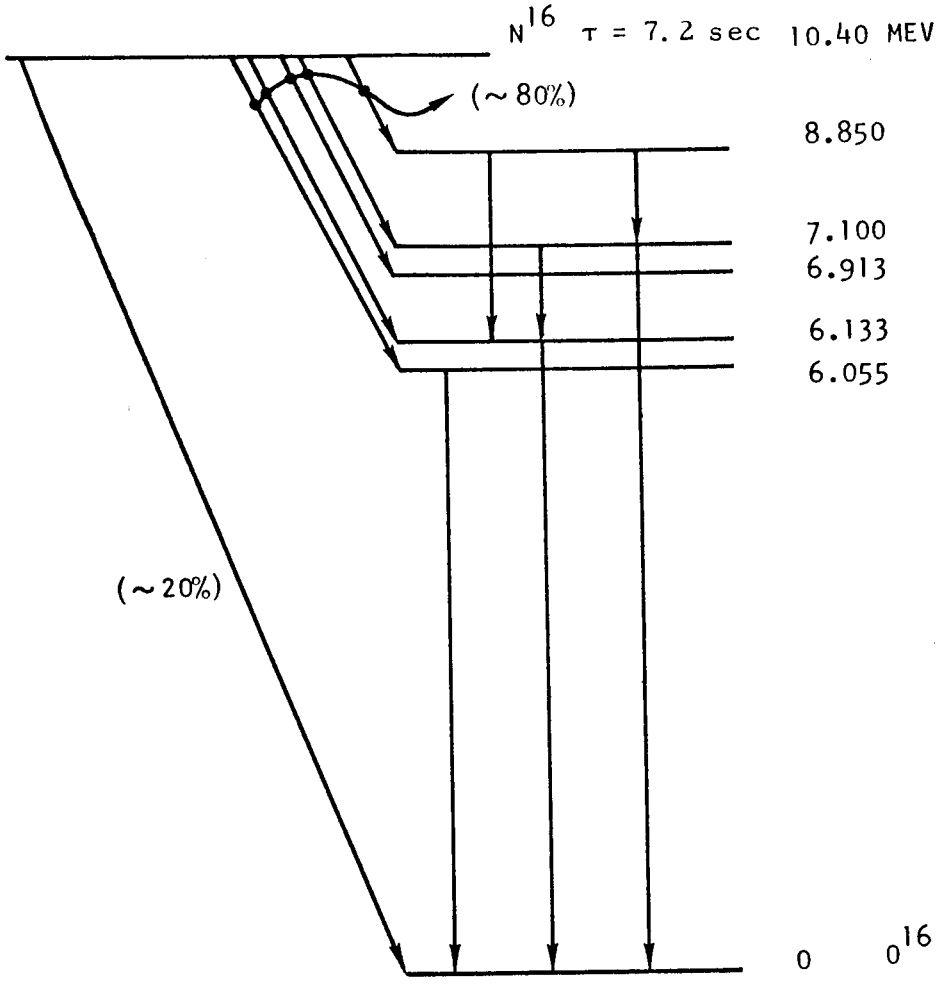

Fig. 1--Decay scheme of N^{16}

completion. This machine is priced at the same level as the one currently being used on Contract NAS3-2537; however, it incorporates several major improvements. First, it is capable of producing beam currents as high as 2.5 milliamp. This is larger by a factor of 2.5 than is presently possible. Since the neutron production is directly proportional to the beam current, the flux (neutrons/cm^2-sec) at the sample position should approach 2.5×10^9 neutrons/cm^2-sec. This represents an increase in sensitivity from 1000 counts per milligram of oxygen to 2500 counts per milligram of oxygen. The second major improvement is the new vacuum system. The new accelerator is equipped with a Vac-Ion pump. The present machine uses a diffusion pump, which allows some oil vapor to enter the accelerating tube. Here it is ionized and builds up on the target surface. As a consequence, target life and neutron production are reduced. Without the carbon, targets last up to 50% longer.

An underground site is being used for the new building. Using a U-shape design, the two ends of the analytical system will now be only 12 ft apart. The travel distance is reduced by a factor of three with this arrangement. Travel times of 1/2 sec should be a routine procedure.

III. EXPERIMENTAL

SAMPLE TRANSFER SYSTEM

Installation of the new dual transfer system has increased the sensitivity of the analytical system for oxygen determinations and at the same time has increased the precision of the method. The improvement in sensitivity was the result of designing the terminal end so that the sample could be closer to the neutron source and decreasing the travel time from the source to the detectors. The new system is approximately five times as sensitive as that in operation at the beginning of the contract. Travel times of 1 sec are now routinely used, producing a sensitivity of over 1000 counts per milligram of oxygen with a new target and full beam power.

Better precision of the method is a result of a more uniform irradiation, due to sample rotation, and a more accurate measurement of the neutron source behavior. The effect of sample rotation is seen in Table 1. The average error of 1.3% for the five determinations is very near the maximum precision possible with this experiment. Counting statistics alone would account for a ±1.1% standard deviation. During this same experiment, the neutron flux was determined with a BF_3 counter. Using these values in place of the monitor standard, an average error of 2.7% resulted.

Table 1

EFFECT OF SAMPLE ROTATION DURING NEUTRON IRRADIATION

Oxygen Added (mg)	Sample Count	Monitor Count	Oxygen Found (mg)	% Error
Rotation of Sample and Monitor [a]				
69.1	23,511	13,220	67.4	2.5
69.1	23,820	12,820	69.4	0.3
69.1	23,760	12,690	70.1	1.4
69.1	23,213	12,730	68.9	0.3
69.1	20,940	11,260	70.5	2.0
				1.3 avg.
No Rotation				
69.1	19,940	11,880	63.6	8.0
69.1	22,730	11,590	74.6	8.0
69.1	23,640	12,260	69.8	1.0
69.1	23,870	13,060	66.5	3.8
69.1	21,860	11,590	71.4	3.3
				4.8 avg.

[a] Sample contained 69.1 mg oxygen as oxalic acid sealed in polyethylene. Monitor was 1/2-in.-diam by 2-in.-long Lucite rod. Both rotated during irradiation.

DETECTOR SYSTEMS

In order to measure small amounts of N^{16} in the presence of large amounts of other activity, a Cerenkov detector was examined, because of previous successes with this system at General Atomic.[2] In those experiments a 3-in. -diameter by 2-1/2-in. -high flat-bottomed glass vessel was filled with water and a glass cover plate was cemented to the top. The device was placed on a 3-in. -diameter DuMont multiplier phototube. The assembly was held together and made light-tight with black tape. It was not shielded. Output from this detector was analyzed with a multichannel analyzer. Samples were sealed in polyethylene vials and activated in a TRIGA Mark I reactor.

The Cerenkov detector was investigated with respect to its response, at several phototube voltages and amplifier gains, to the beta radiations of N^{16} and Cl^{38}. The same isotopes were also examined with a standard NaI(Tl) detector as a comparison.

The sensitivity of detection of N^{16}, from the reaction $O^{16}(n, p)N^{16}$, varied with counting conditions as shown in Table 2. Activities shown in the table are corrected to counts per minute per gram of oxygen at the end of the irradiation. Data are shown for certain groups of channels as well as for the total activity in all but the lowest four channels. The detector has the background characteristic shown in Table 3.

Two sets of three oxygen standards each were run in duplicate and the specific N^{16} activity was compared with that of the water standard. The results are shown in Table 4.

The spectra of Cl^{38} and N^{16} obtained with the Cerenkov detector are given in Fig. 2. Three spectra are shown: one from 49.5 mg of chlorine as the ammonium salt; one from 48.5 mg of chlorine as NH_4Cl in 0.964 g of water; and one from 1.0732 g of water. The Cl^{38} activity level 3 min after irradiation was 98 mr/hr at 1 cm. The spectrum from Cl^{38} terminates at channel 34. Counts in channels ≥ 35 from the Cl-H_2O sample were taken to be due to N^{16} activity produced during irradiation via the $O^{16}(n,p)N^{16}$ reaction. Calculation of the activity in channels ≥ 35 from both H_2O and H_2O-Cl samples showed a sensitivity of 6.30×10^4 cpm/g and 6.15×10^4 cpm/g of oxygen, respectively.

Examination of the H_2O-Cl sample was also attempted with a 3 in. by 3 in. NaI(Tl) detector using the same irradiation conditions. "Pileup" in the detector rendered it useless for this analysis.

For the current problem, that of oxygen in potassium, a Lucite cylinder was fitted with two 3-in. multiplier phototubes. A hole was drilled

Table 2

CPM N^{16} PER GRAM OF OXYGEN WITH CERENKOV COUNTER AFTER 0.25 MIN IRRADIATION IN THE TRIGA MARK I REACTOR

Multiplier Phototube Voltage (v)	Amplifier Gain	Absorber	CPM per Gram of Oxygen				Total Specific Activity
			Channels 5-9	Channels 10-19	Channels 20-29	Channels >29	
900	1/4		8.0×10^{5}	1.3×10^{4}	3.0×10^{3}	2.3×10^{3}	8.2×10^{5}
900	1		3.0×10^{6}	8.6×10^{5}	8.1×10^{5}	1.5×10^{5}	4.8×10^{6}
900	1	Polyethylene, 1/2 in.	1.07×10^{6}	6.9×10^{5}	2.5×10^{5}	3.8×10^{4}	2.1×10^{6}
900	1	Aluminum, 3/4 in.	4.5×10^{5}	3.6×10^{5}	1.9×10^{5}	2.7×10^{4}	1.0×10^{6}
1100	1/4		1.9×10^{7}	3.0×10^{6}	1.4×10^{6}	7.6×10^{5}	2.4×10^{7}
1100	1/2		6.2×10^{7}	2.0×10^{7}	2.6×10^{6}	3.2×10^{6}	8.8×10^{7}

Table 3

CERENKOV COUNTER BACKGROUND

Phototube Voltage (v)	Amplifier Gain	Channel Groups (cpm)				Total cpm
		5-9	10-19	20-29	>29	
900	1/4	36	32	0	0	68
900	1	5,156	92	8	4	5,260
1,100	1/4	50,080	2,568	20	4	52,672

Table 4

OXYGEN DETERMINATION IN STANDARDS

Standard Sample	Multiplier Phototube Voltage (v)	Amplifier Gain	Average Oxygen (wt-%)	Actual Oxygen (wt-%)
Graphite, [a] 1	900	1	2.42 ±0.07	2.54
Graphite, 2	900	1	1.03 ±0.03	1.06
Graphite, 3	900	1	0.28 ±0.02	0.29
Mineral oil, [b] 1	1100	1/4	0.125 ±0.010	0.16
Mineral oil, 2	1100	1/4	0.652 ±0.012	0.664
Mineral oil, 3	1100	1/4	1.130 ±0.040	1.14

NOTE: All samples were sealed in polyethylene.
[a] Standardized with NaI detectors.
[b] Oxygen added as oxalic acid.

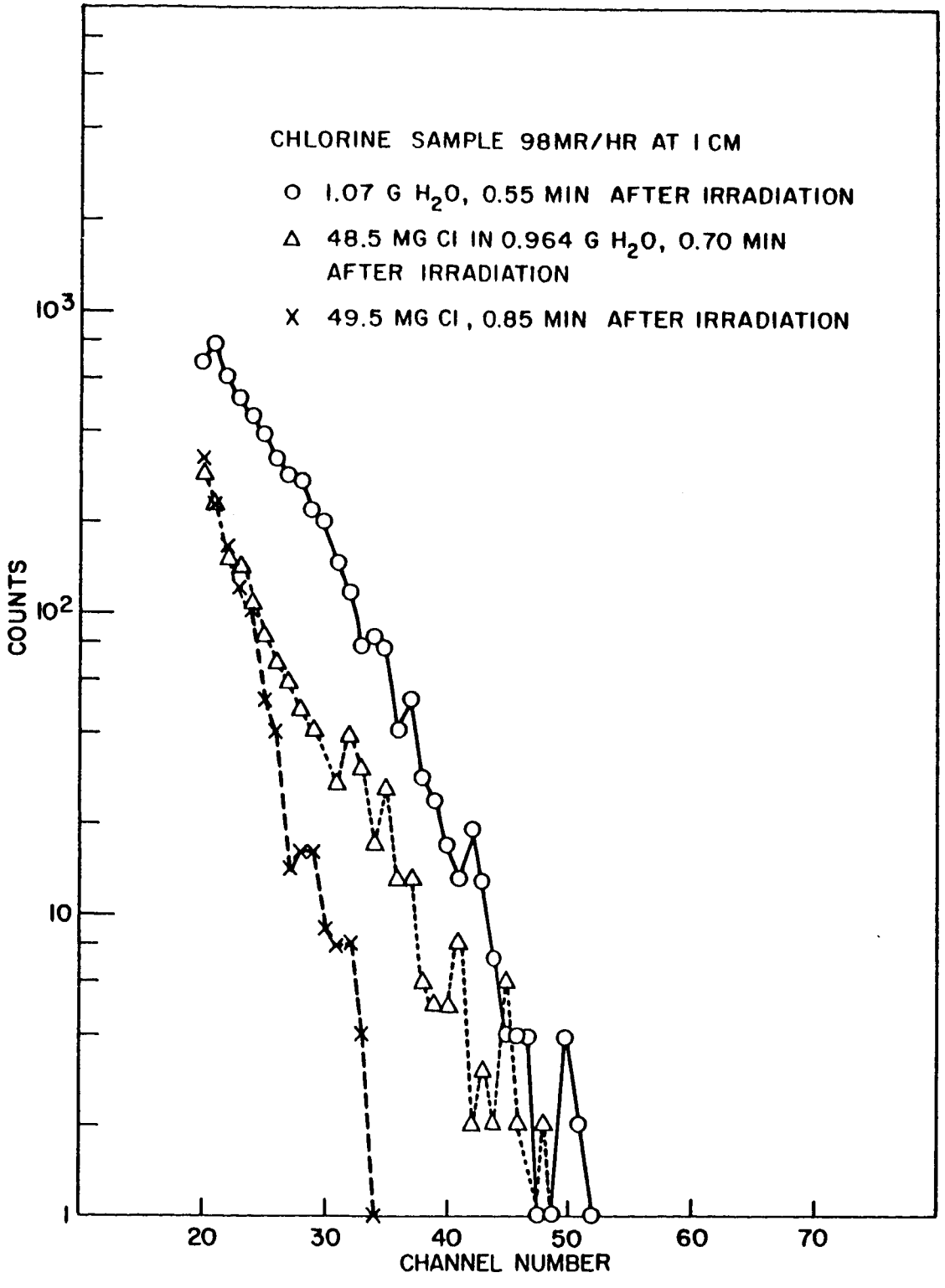

Fig. 2--Effect of 98 mr/hr Cl^{38} level on N^{16} spectrum, using Cerenkov counter

in the center to accommodate the transfer tube. The concept of two photo-tubes allows the utilization of coincidence electronic circuitry, which eliminates almost all electronic "noise." Although the investigation of this detector system is still in the preliminary stages, sensitivities of better than 500 counts per milligram of oxygen have already been obtained, even in a gamma-ray background of 50 mr/hr.

SAMPLE ENCAPSULATION

A variety of metals and alloys have been examined for their oxygen contents in an effort to find a capsule material that would reduce the blank value. Some of the materials were vacuum degassed, some were sanded with silicon carbide, some were filed, and some were heated in a hydrogen furnace. All samples were irradiated with 14-Mev neutrons for 20 sec and counted for 30 sec with a single-channel or a multichannel analyzer. Results of these investigations are listed in Table 5. The inert-atmosphere "dry box" under construction should reduce these blank values by a factor of two.

IV. CONCLUSION

The third quarter of the contract period has been devoted mainly to construction and assembly of an analytical neutron activation analysis system capable of precision, accuracy, and sensitivity which reflect the current state of the art. Parts of the system which represent a significant improvement are:

1. The simplified control panel, which automatically operates the transfer system, the accelerator, and the analyzers. This eliminates the need for a highly trained technical analyst.

2. The dual transfer system, which compensates for any variation in neutron production during an irradiation.

3. The single-channel analyzer, which eliminates the need for a printout device and spectrum summing.

4. The Cerenkov detector, which eliminates the need for extensive shielding and reduces "pileup" problems associated with sodium iodide scintillation counting.

In the examination of possible metal containers, OFHC copper was found to be superior to aluminum. It has a low oxygen content, is easier

Table 5

OXYGEN CONTENT OF POSSIBLE CAPSULE MATERIALS

Sample	Weight (g)	Sample Treatment	Oxygen Content	
			mg	ppm
OFHC copper	23.65	None	2.85	120
OFHC copper	22.43	Hydrogen furnace	0.206	9.2
OFHC copper	20.20	SiC sanding	0.667	33
OFHC copper	20.83	Filing	1.23	61
Brass	38.52	Vacuum furnace	3.51	91
Steel	43.30	Vacuum furnace	3.16	73
Molybdenum	15.18	Vacuum furnace	2.21	145
Molybdenum	14.95	Vacuum furnace	1.82	122
Niobium	5.18	Vacuum furnace	0.50	96
Niobium	5.21	Vacuum furnace	0.49	94
Steel	34.81	Zone refined	0.275	8
Steel	34.81	Zone refined	0.335	4
Steel	34.81	Zone refined	0.222	6

to clean, can be sealed by pinching or welding, and requires no machining. With the proper handling techniques, concentrations of less than 5 ppm oxygen should be present in the blank. With a capsule weight of 2 g or less, the total oxygen blank would be under 10 μg.

V. FUTURE WORK

Samples of potassium metal are being encapsulated by NASA-Lewis as well as several NASA contractors. The oxygen content of these materials will be determined. Separate oxygen determinations on these samples have been made using the mercury amalgam method. A standardization of the activation analysis method will be made using a single potassium sample and the technique of standard additions.

Engineering drawings or electronic circuit diagrams of all equipment will be submitted with the final report.

VI. REPORTABLE ITEMS OF ARTICLE 24 DURING REPORT PERIOD

During the period of this report the following "reportable items," as defined by Article 24, "Reporting of New Technology," evolved:

NONE

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1. Belcher, E. H., Proc. Roy. Soc., A213, 90 (1953).
2. Lukens, H. R., and Lasch, J. E., "Use of the Cerenkov Counter in the Determination of Oxygen by Neutron Activation Analysis," General Atomic Report GA-3837, January 15, 1964.

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